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# High Temperature Heterogeneous Materials

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Final Technical Report

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## 1. Executive Summary

The objective of the research, reported on here, was to develop experimental and supporting analytical methodologies for the determination and assessment of the integrity of interfaces in composite materials at both room and high temperatures. The experimental test techniques developed in this program are described and supporting analytical methods to assist in the determination of interface fracture toughness from experimental measurements are presented. The test techniques consist of the tensile test on a thin coated fiber and four point bend of a bimaterial bar and were chosen on the basis of their suitability for use at high temperatures. A key goal in the analytical work was to develop analytical and numerical tools for the analysis of film cracking (or matrix cracking in the case of fiber-reinforced composites) which do not employ the simplifying assumptions typically made in shear lag and stress-transfer models. The analytical methods include, for the first time, a fracture mechanics approach to the determination of interface fracture toughness from experimental measurements of crack spacing, a rigorous treatment of matrix cracking in fiber-reinforced composites and an analysis of the stress fields around a crack terminating at a frictional interface. The experimental testing techniques, together with the supporting analytical developments, described in this report lay the foundations for a ready transition to the treatment of interface phenomena at high temperatures.

In addition to the above-mentioned investigations of interface phenomena in composite materials, an experimental study of crack tip shielding/antishielding by impurities in alpha-silicon carbide was undertaken. This investigation required the application of stress during high temperature (1873 K) anneals. A novel stress fixture, based on thermal expansion mismatch, was designed, constructed and utilized to achieve this in-situ stressing of the crack specimens at high temperatures.

## 2. Introduction

The goals of the research project, together with some of the key accomplishments were summarized in Section 1 above. In the following sections, these accomplishments are discussed in more detail. In Section 3, the experimental and analytical investigations on interface cracking phenomena are described. The experiments devised and carried out on this program are discussed in Section 3.1 along with some supporting analytical developments. Supporting analytical techniques for the study of interface phenomena are described in Section 3.2. The development of domain integrals for the computation of interface fracture parameters in axisymmetric geometries is described in Section 3.2.1. Analysis techniques, developed in this program, for multiple cracking phenomena are discussed in Section 3.2.2. One of the key objectives of this facet of the research was to develop numerical and analytical tools to study these multiple cracking phenomena without recourse to the simplifying assumptions typically employed in shear lag or stress transfer models. This objective has been satisfied in several ways. In Section 3.2.2 a description of a rigorous analytical approach to matrix cracking in fiber-reinforced composites, based on linear elastic fracture mechanics and Love's stress functions, is reported on. The effects of various degrees of interfacial adhesion and slip are also examined. In the same section, a computational fracture mechanics approach to the analysis of multiple cracking phenomena in brittle films on dissimilar planar and axisymmetric elastic substrates is outlined. A methodology for the determination of film (or matrix) fracture toughness from film crack densities has been developed and is briefly outlined. A procedure for the determination of interface fracture toughness from crack densities has also been developed. In Section 3.2.3, the application of Mellin integral transforms to the study of a crack terminating at a frictional interface between two materials is described, while in Section 3.2.4, a procedure for the determination of stress fields of phase boundary dislocations is discussed.

Section 4 describes the experimental study of crack tip shielding/antishielding. The underlying crack theory is briefly addressed in Section 4.1, while the experiments are described in Section 4.2.

References cited in the text of the report are given in Section 5., followed by Figures in Section 6. Presentations made by faculty on their work on this research project are listed in Section 7. A list of publications, emanating from research which was either fully or partially supported by the research grant, is given in Section 8. A set of publications accompanies this report.

### **3. Interfacial Phenomena**

#### **3.1 Experimental ( K. T. Faber and A. Cazzato )**

A test technique was developed to measure interfacial shear strength through observation of crack densities in bimaterial concentric cylinders. In the bimaterial rod experiment, concentric cylinders were chosen as an accurate representation of the fiber-matrix geometry where a rod of "fiber" material is coated with a thin (50 - 500  $\mu\text{m}$ ) film of "matrix" material. When the rod is pulled in tension, many cracks form in the film and the spacing of the cracks can be related to the mechanical properties of the interface. The first system studied consisted of a sodium-silicate brittle coating on an aluminum rod. A polymeric interphase layer was added for comparison to the specimens with unmodified interfaces.

If the coating strength was perfectly uniform over the length of the specimen, the saturated crack pattern would be composed of equally spaced cracks. However, because of the statistical distribution of flaws and flaw sizes, the coating strength is not single-valued. For coatings with non-single-valued strengths, higher crack densities and less uniform array of crack spacings is expected. Bending strengths of the matrix material were used to gather an independent measure of the variability of sodium silicate matrix properties and were described using a two-parameter Weibull distribution.

A computer program was written to incorporate the statistical nature of strength into calculations of interface strength from crack density. The distribution of strengths was modeled by a Monte Carlo simulation based on an ultimate strength criterion, i.e. coating cracks formed when the local normal stress exceeded the local strength. Likewise, debond cracks grew when the local interfacial shear stress exceeded the shear strength of the interface. Both normal and shear stresses were calculated by a shear lag analysis.

Bimaterial specimens having both modified and unmodified interfaces showed a saturation with increasing applied stress as shown in the crack density vs. strain profiles in Figure 1. The variation in strength of the coating ("matrix") material is shown in Figure 2. By fitting the failure strengths to a two-parameter Weibull distribution, the Weibull modulus, corresponding to the best fit line, was found to be 1.9 - characteristic of a broad distribution in strengths. Computer simulation results in Figure 3 show the trends in crack density as a function of Weibull modulus and interfacial shear strength. Each curve represents a single Weibull modulus coating for a variety of interfacial strengths simulated for material properties representative of the aluminum- sodium silicate system. As expected, the crack density increases with interface strength. More importantly, the crack density increases appreciably with decreasing Weibull modulus. In order to establish the interfacial shear strength for the present experimental studies, the multiple cracking simulation was run with experimentally determined Weibull parameters. For comparison, nearly single-valued coating strength (Weibull modulus = 100) also was simulated. These results are shown in Figure 4 where interface strength is plotted as a function of crack density. Experimental crack density measurements are shown as dotted lines on the graph indicating that shear strengths were determined to be approximately 0.7 MPa for the unmodified interface and 0.5 MPa for the modified interface. The assumption of a single-valued coating ("matrix") strength would lead to a five-fold overestimation of the ultimate shear strength. Hence, the importance of strength statistics is clearly established.

Other systems where this test has been examined include  $\text{Al}_2\text{O}_3\text{-ZrO}_2$ , glass-SiC, and  $\text{Al}_2\text{O}_3$ -glass. The fracture mechanics approach to multiple cracking by Moran and Nahta described in a subsequent section in this report will provide further insight into the cracking behavior we have observed.

In a second fracture mechanics approach, crack opening displacements have been measured on the concentric cylinder test of a  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  specimen containing a single annular crack. No debonding was observed prior to failure. Bimaterial bend specimens of alumina-alumina and alumina-Sapphire have been tested in four point bending to examine interfacial fracture toughness. Existing analytical and finite element results for isotropic and transversely isotropic pairs were used utilized in analysis of this data.

### 3.2 Analysis (L.M. Keer, B. Moran, T. Homulka and R. Nahta)

The objective of this area of the research project was to provide the analytical and numerical input to the development and understanding of the coated fiber multiple cracking experiment being carried out by Professor K. Faber. This experimental configuration was chosen on the basis of its suitability for use at high temperatures. In recent years, an interface fracture mechanics methodology has emerged in which interfaces are characterized on the basis of interface fracture energy or interface toughness. This approach is particularly suitable for ceramic systems. Several investigators have analyzed such systems in the past by assuming that the integrity of the interface can be modeled using an interface shear strength criterion. Simplified shear-lag or stress-transfer models are then typically used to determine the interface shear stress distribution and that value at the critical load taken to be the strength of the interface. In effect, shear-lag and stress-transfer models reduce the singular stress field at a fiber-matrix interface to a finite value. The magnitude of this finite value depends on the approximations made in the model in question and can differ significantly from model to model. (In elastic-plastic systems or in fiber pull-out problems with a steady frictional stress these models may be appropriate).



For these reasons we have pursued a purely fracture mechanics approach to the problem of multiple cracking and the determination of interface fracture characteristics. Several new results have emerged from our research in this area:

#### 1. Domain integrals.

Development of a domain integral methodology for evaluation of energy release rates, stress intensity factors and phase angles in numerical modeling of axisymmetric crack geometries - such as fiber pull-out problems or the coated fiber experiment (Nahta and Moran, 1993a)

#### 2. Multiple Cracking Phenomena

a) Fracture mechanics based analysis of multiple cracking in coatings. Determination of coating fracture toughness from crack spacing in well-bonded coatings (Nahta and Moran, 1994)

b) Fracture mechanics based analysis of simultaneous matrix cracking and interface debonding in multiple cracking of coated fiber. Determination of interface toughness from crack spacing (Nahta and Moran, 1993b)

### 3.2.1 Domain Integrals

At the outset of the research program we observed several shortcomings in the numerical tools available for the accurate and efficient determination of interface fracture parameters (such as mixed mode energy release rate  $G$ , the stress intensity factors  $K_I$  and  $K_{II}$ , and the mode mixity phase angle  $\psi$ ) in the axisymmetric geometries of interest. Accordingly we developed a domain integral based methodology (Nahta and Moran, 1993a) to alleviate these shortcomings. The method is particularly suited for fiber disbond or pull-out problems and for matrix cracking in coated fiber configurations. For the extraction of the individual stress intensity factors, this approach relies on specification of the plane auxiliary fields. Since the plane strain fields are imposed over the curvilinear frame, both the equilibrium and the compatibility of the auxiliary fields are not satisfied,

thus resulting in additional terms which do not arise in plane problems. Previously, Charalambides and Evans (1989) studied the problem of interface debonding in a fiber pull out test. They employed the stiffness derivative method to compute energy release rates and phase angles and noted that difficulties arise in the specification of auxiliary fields in the extraction of mixed mode stress-intensity factors. Furthermore, their formulation only allows the specification of the auxiliary fields in the immediate vicinity of the crack tip which may lead to a loss of accuracy and/or required a very fine mesh around the crack tip. This difficulty does not exist in the domain integral formulation since the auxiliary fields are specified over the entire domain. This approach is found to be a very accurate and efficient method and can be readily implemented into the post processing stage of any standard finite element (boundary element, etc.) program. In Figure 5, the normalized energy release rate  $\frac{E_f G}{\sigma^2 t}$  is shown as a function of debond crack length  $d/t$ . The steady state portion of the curve is seen to be in excellent agreement with the analytical solution (i.e., where  $G/G_{ss} = 1$ ). The steady state conditions are simultaneously reached in normalized energy release rate  $\frac{E_f G}{\sigma^2 t}$  and phase angle  $\psi$  (not shown here).

Finally, this approach can be extended to the case of a general curvilinear crack front in three dimensions and would be useful for the determination of fracture parameters along a surface breaking interface crack, for example.

### 3.2.2 Multiple Cracking Phenomena

The multiple cracking phenomenon observed in tensile experiments on thin coated fibers (Lowden and Stinton, 1988; Ochai and Murakami, 1990; Cazzato and Faber, 1993) is examined from a fracture mechanics perspective. Such experiments are used to characterize both coating and interface strength or integrity. Other experiments based on the same principal, include tensile tests on unidirectional fiber composites and tensile or residual stress induced cracking of thin films. While not the primary focus of this project, we

expect that the analytical methodology developed here will be directly applicable to these situations also.

As mentioned above, we have chosen to characterize the integrity of the interface in terms of its fracture toughness. For an imperfect interface, the saturated crack spacing observed in the experiments can be explained in terms of the competition between the processes occurring simultaneously namely the interface debonding (adhesive failure) and matrix cracking (cohesive failure). In a fracture mechanics approach, the failure processes are described in terms of energy release rates. For example, an interface crack is expected to grow when the crack driving force, expressed in terms of the energy release rate  $G_i$  is equal to the interface fracture toughness  $\Gamma_i$ . Similarly, a matrix crack is expected to occur when the matrix energy release rate  $G_m$  equals the matrix fracture toughness  $\Gamma_m$ .

The fracture quantities which are required, and which must be obtained analytically or numerically, are the matrix energy release rate  $G_m$  and the interface debond energy release rate  $G_i$  as a function of segment length and debond crack length. We employ the axisymmetric domain integral methods (Nahta and Moran, 1993a) in conjunction with the finite element method to compute these fracture quantities.

The interface fracture/debonding plays an important role in determining the matrix crack spacing. In the experiment, matrix cracking occurs at some critical value of the load and as the load is increased further, the matrix crack spacing decreases until it eventually saturates. In our analysis, we use the case of a perfect interface as a reference. In the absence of interface debonding (Perfect Interface,  $\Gamma_i = \infty$ ), the crack spacing continues to decrease as the applied stress is increased and no saturated crack spacing is observed. A weak interface leads to wider crack spacing whereas for a stronger interface, the crack spacing observed would be smaller, but still larger than that observed for a perfect interface.

### *Matrix Cracking in a Fiber-Reinforced Composite with Slip at the Fiber-Matrix Interface*

A linear elastic fracture mechanics analysis of interfacial slip under longitudinal tensile loading in a fiber reinforced brittle matrix composite with matrix cracks terminating at the interface is presented (Fig. 6). The present analysis does not employ any of the simplifying assumptions used in shear-lag analyses such as where the displacement in the radial direction in the matrix is assumed to have a prescribed distribution. The problem is formulated using Love's stress functions and two coupled singular integral equations are solved to determine the two basic unknowns which are related to the crack opening displacement and the gradient of the interfacial slip when a constant shear stress distribution is assumed in the slip region.

The interfacial adhesive shear stress is determined for different ratios of shear moduli, fiber volume fractions and slip lengths. Complete stress fields are obtained for a brittle matrix fiber-reinforced composite, calcium aluminosilicate glass ceramic reinforced with silicon carbide fibers (SiC/CAS), and are compared with the case when there is perfect adhesion at the interface

### *Multiple Cracking in Coating - Perfect Interface*

The case of a perfect interface is of some significance in its own right. For a strong coating, little or no interface debonding occurs and thus the crack spacing observed is controlled by the coating fracture toughness. Thus, for strong interfaces, this experimental configuration can be used to determine matrix or coating toughness. Thouless (1990) analyzed this problem for a planar geometry and for homogeneous materials. Here we have extended the analysis to account for axisymmetric as well as planar geometries and for bimaterial systems where the coating properties differ from those of the substrate or fiber (Nahta and Moran, 1994).

### *Finite Element Analysis of Matrix Cracking with Interface debonding*

A finite element analysis of multiple cracking with interface debonding has been carried out. The paper by Nahta and Moran (1993b) describes the analysis procedure and results in detail. The objective of this study is to determine interface fracture toughness from observed matrix crack spacing (segment lengths) allowing for interface debonding.

Figure 7 shows the minimum crack spacing  $l/t$  as a function of applied stress  $\sigma$  for various values of the interface fracture toughness  $\Gamma_i$ . The lower curve corresponds to the case of perfect interface ( $\Gamma_i = \infty$ ) for which no interfacial debonding occurs. As expected, all other crack spacing curves shift to the right and upward of this curve, since the crack spacing increases with a decrease in the interface fracture toughness. In addition, matrix cracking does not begin until the stress reaches a critical stress value called matrix crack initiation stress  $\sigma^*$ . At the occurrence of the first matrix crack, if the interface is sufficiently weak (its interface fracture toughness is smaller than a specific value  $\Gamma_{\min}$ ), it will completely debond. For stronger interfaces ( $\Gamma > \Gamma_{\min}$ ), the coated rod will undergo further matrix cracking with simultaneous partial debonding of the resulting segments until eventually a saturated crack spacing is reached. For example, for long segments, the saturated crack spacing is attained when the interface debonding has reached a steady state (continues without increase in stress) therefore preventing any further matrix cracking. Note that when the interface debond crack is not in steady state, it just delays the matrix cracking.

As the interface fracture toughness increases, the crack spacing curve approaches that for a perfect interface. Note that the debonding is initiated at that applied stress where the crack spacing curve deviates from the curve for a perfect interface. Figure 8 shows a cross plot of saturated minimum crack spacing,  $l/t$ , as a function of interface fracture toughness  $\Gamma_i/\Gamma_m$ . This curve allows us to determine the interface fracture toughness for a given saturated crack spacing which is of particular interest in the experimental

determination of fracture toughness. From this curve, the interfaces can be classified into 3 categories namely weak, intermediate and strong. As already noted, weak interfaces are those whose fracture toughness  $\Gamma_i$  is lower than a specific value  $\Gamma_{\min}$  and will completely debond at the onset of matrix cracking. In the intermediate range ( $\Gamma_{\min} < \Gamma < \Gamma_{\max}$ ), the crack spacing is sensitive to the interface fracture toughness and quantitative results (fracture toughness values) can be obtained from the experiments. Finally, strong interfaces ( $\Gamma > \Gamma_{\max}$ ) behave essentially as perfect interfaces over a significant load range. Thus the experiment yields qualitative information on weak and strong interfaces and quantitative information on interfaces in the intermediate range.

The fracture mechanics approach employed in this study is an innovative approach and offers a clear explanation of multiple matrix cracking and saturated crack spacing and relates the latter quantity to the interface fracture toughness. The approach is computationally intensive, however, and requires many detailed finite element calculations for the evaluation of matrix energy release rate  $G_m$  and interface energy release rate  $G_i$  for a range of material and geometric parameters. We are currently exploring the development of simple fracture mechanics based models of this process.

### **3.2.3 Stress Fields Around a Crack Tip Terminating at a Frictional Interface Between Two Materials**

The problem of a crack terminating at an interface (see Fig. 9) between two materials which is governed by Coulomb's law of friction is studied using Mellin integral transforms (Wijeyewickrema and Keer, 1993; Wijeyewickrema, Dundurs and Keer, 1993). Depending on the relative slip directions of the two wedges that are created by the crack, both the case of the two wedges moving in opposite directions and that of the two wedges moving in the same direction are treated. The characteristic equations which yield the order of the crack tip singularity are obtained in terms of the Dundurs constants, the inclination of the crack and the coefficient of friction. For the special case when the crack is

perpendicular to the interface and the two wedges slip in opposite directions, it is shown that the problem decouples into mode I (symmetric) and mode II (antisymmetric) cracks, and numerical results are presented for this case.

### **3.2.4 Displacement Field for Triangular Phase Boundary Dislocation Loop**

The displacement field of a triangular phase boundary dislocation loop is obtained (Keer and Polonsky, 1993) in explicit form for convenient applications (such as interface fracture mechanics). An accurate procedure for determining the elastic fields of phase boundary dislocations in one of the phases, once those in the other phase are known, is derived.

## **4. Crack Tip Shielding/Antishielding**

### **4.1 Crack Theory (J. Weertman)**

Exact solutions have been found to the problems of the modes I, II and III cracks in a linear hardening solid in general yielding. A unique result in the solution of the mode I crack is that no dislocations are emitted from the crack faces or the crack tips into the interior of the solid. A possible asymptotic solution has been obtained for a mode I crack in small scale yielding in an elastic perfectly plastic solid. General relationships have been obtained (by the graduate student J.A. Hurtado) that relate the nine components of the general non-redundant dislocation density field to derivatives of the nine components of the stress field, including the three rotation pseudo-stress components.

### **4.2 Experimental Study of Crack Tip Shielding/Antishielding by Impurities in Alpha-Silicon Carbide (J. Weertman, K.A. Forland, K.T. Faber)**

In this study, a newly proposed mechanism for toughening ceramics for high temperature structural applications was investigated. In order to facilitate the experimental procedure, an innovative apparatus for testing materials at elevated temperatures was designed, constructed and successfully utilized.

The theory of crack tip shielding/antishielding by impurities was investigated using a commercially available, fully dense  $\alpha$ -SiC. The theory states that a crack tip will be shielded (or antishielded) by a non-uniform distribution of particles in the vicinity of the crack tip. Attempts were made to obtain a non-uniform distribution of  $B_4C$  precipitates in the SiC matrix. In order to achieve this, a tensile stress was applied to pre-cracked SiC specimens during 1873 K anneals. A uniform distribution of precipitates was obtained in specimens during 1873 K anneals without applied stress. Evidence of  $B_4C$  precipitates was observed in the latter specimens using transmission electron microscopy.

In order to stress specimens in situ during the 1873 K anneals, a unique stress fixture was developed. An unusual approach to stress application was necessary because the furnace set-up placed severe constraints on any standard stress-application method. The stress fixture, which took advantage of the thermal expansion mismatch between SiC and alumina, was compact, inexpensive and reusable. In addition, the device allowed the user to set the stress level for each specimen individually. This was particularly useful because specimen dimensions and pre-crack lengths varied from specimen to specimen.

Fracture toughness tests were performed on as-received, heated and heated, stressed specimens of the double cantilever beam configuration. No differences in fracture resistance were observed between the three specimen types, i.e., as-received, heated, and heated, stressed. Theoretical calculations were then performed for comparison to these experimental results.

Calculations based on data from the literature indicate that  $B_4C$  particles in the  $\alpha$ -SiC matrix are undersized, i.e., a negative misfit exists. Precipitation in the tensile field surrounding the crack tip is likely therefore to be inhibited. Calculations following the Weertman-Hack theory show a very small anti-shielding effect for a precipitate-poor region surrounding the crack tip. No effect was observed in the experiments.



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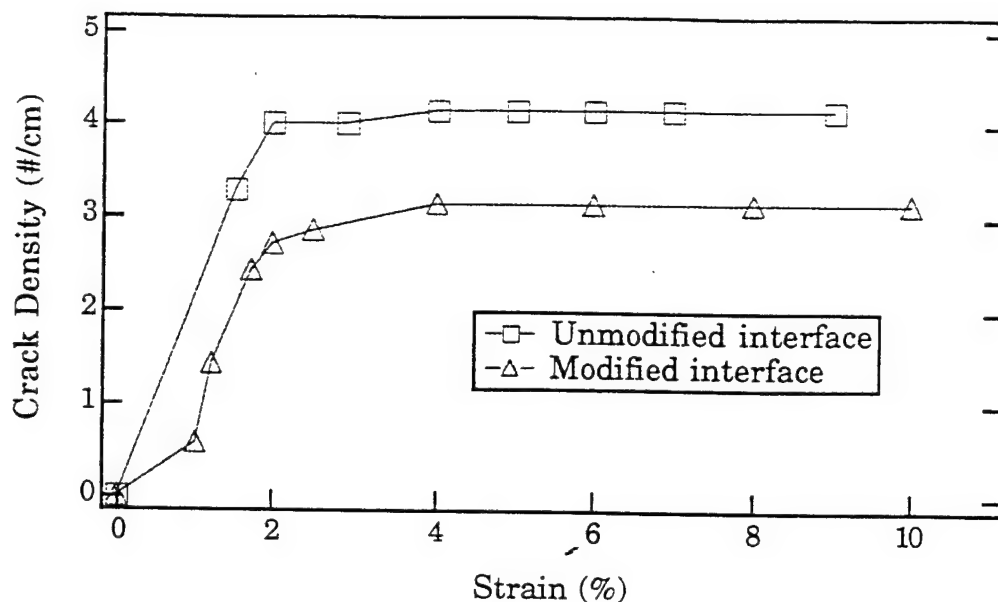


Fig 1: Crack density versus applied strain for two Al-cement concentric cylinders, one with a modified interface, both showing crack density saturation.

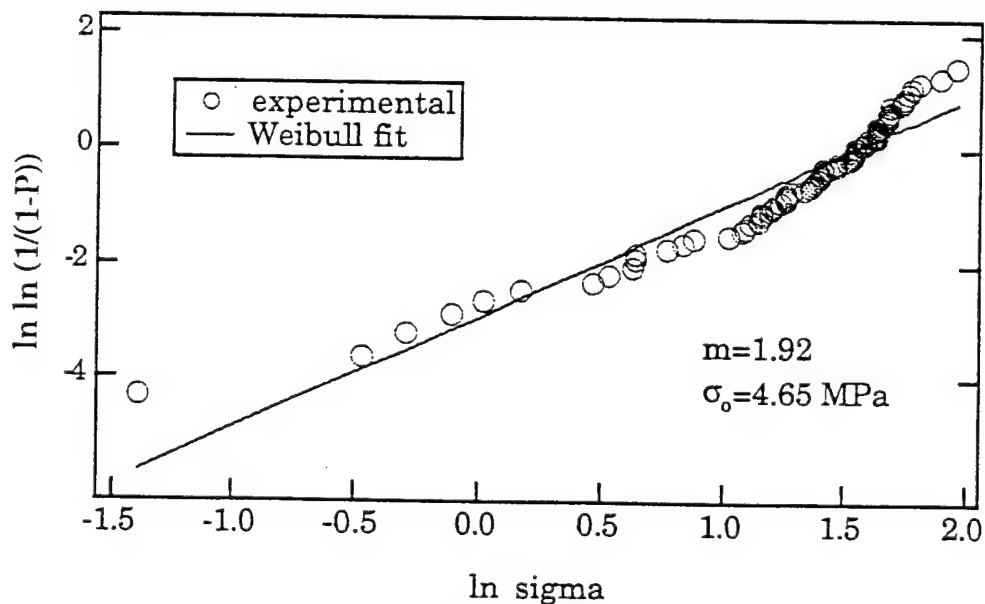


Fig 2: Weibull analysis of four point bend strengths of monolithic soda-silicate cement bars.

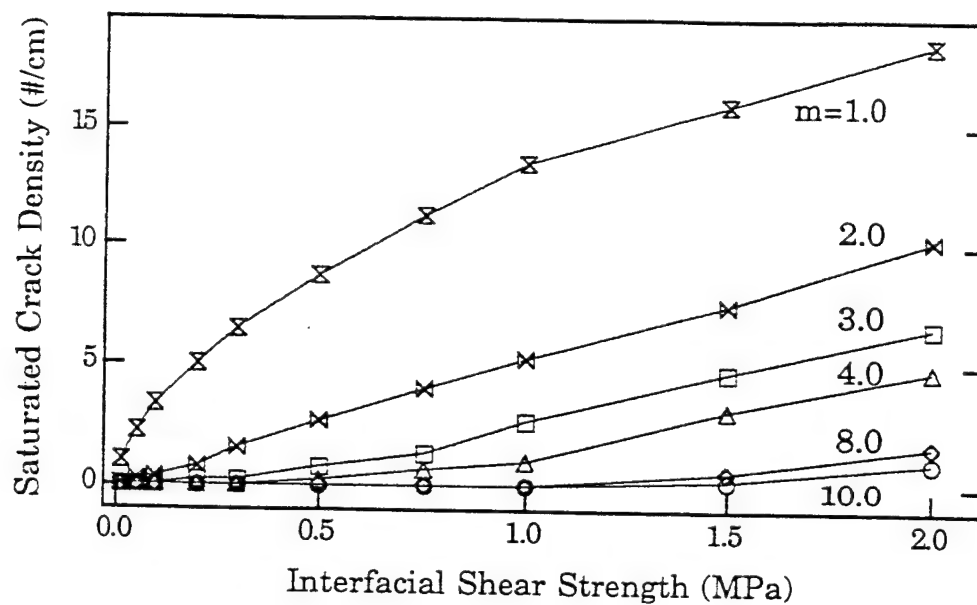


Fig 3: Simulated crack densities as a function of interfacial shear strength and coating Weibull modulus for Al-cement concentric cylinders.

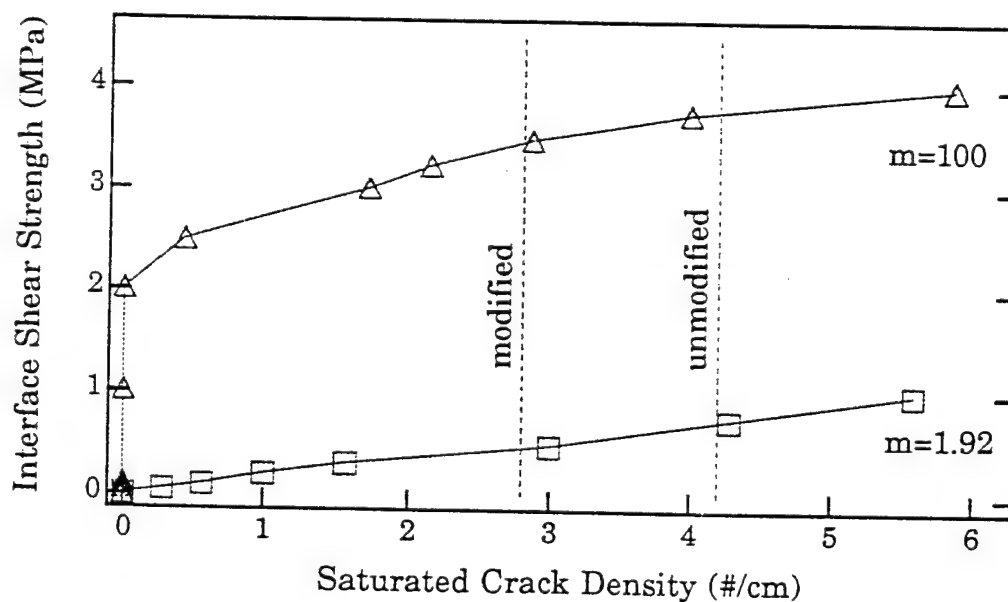
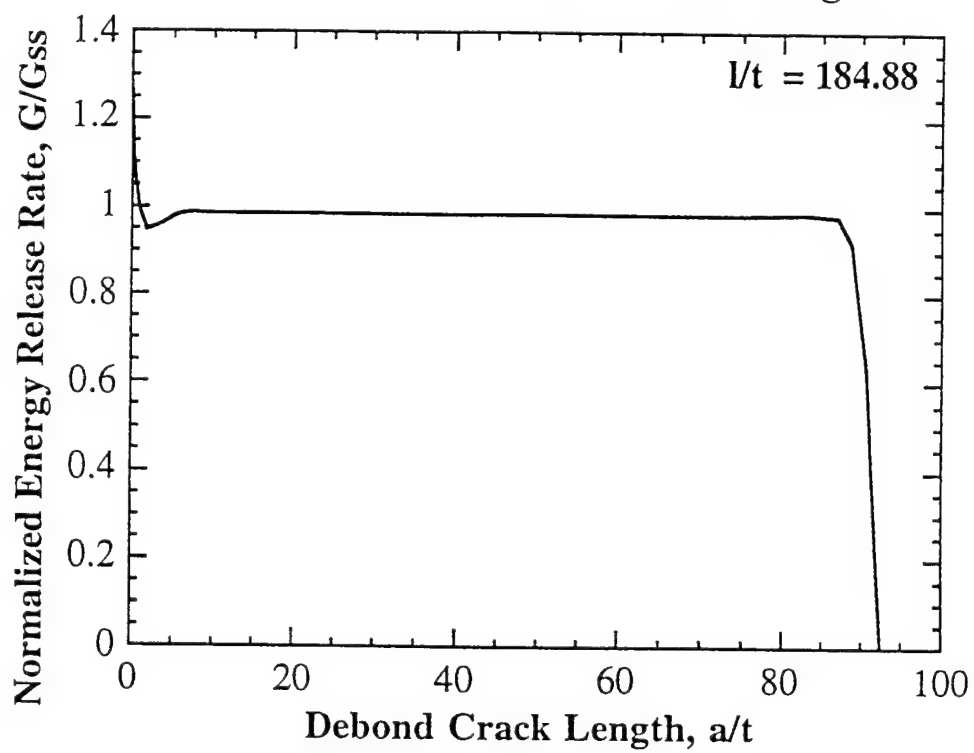
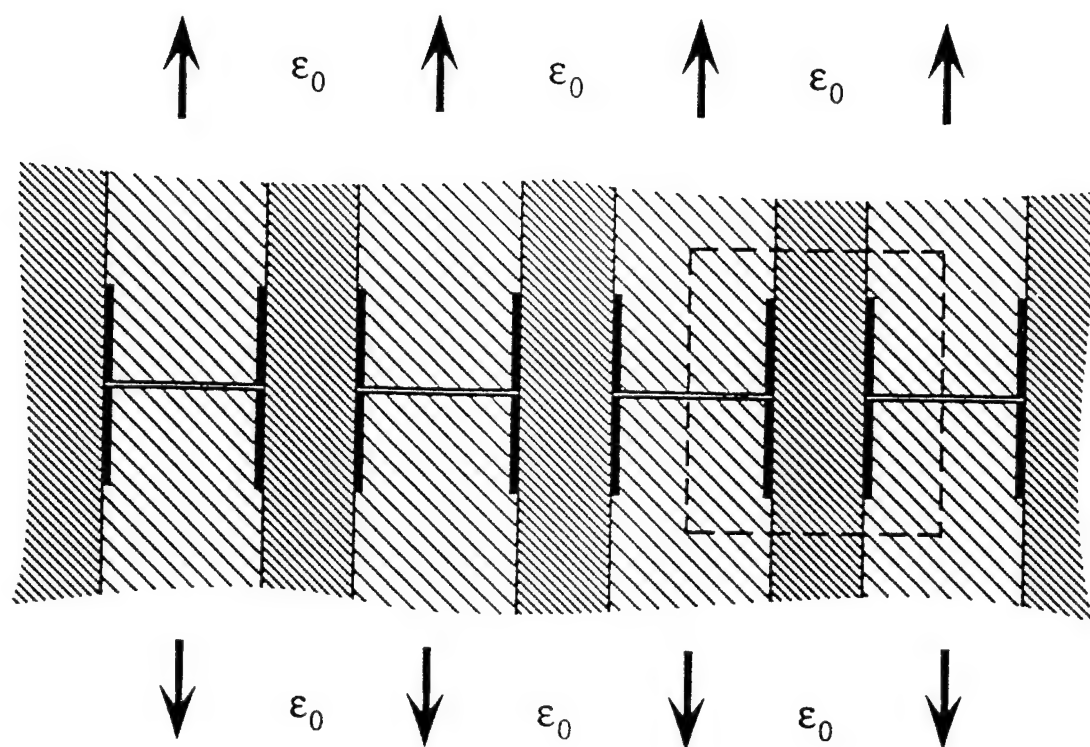


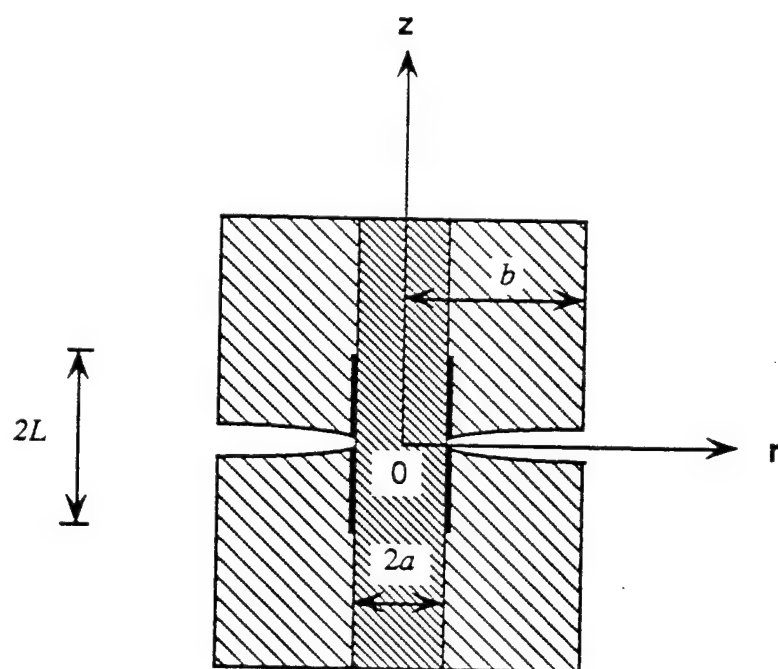
Fig 4: Interfacial shear strength versus simulated crack density for coating Weibull moduli of 1.92 and 100.

**Fig. 5** Interface Energy Release Rate as a Function of Debond Crack Length





(a)



(b)

**Fig. 6** Schematic of Matrix Cracking with Interfacial Slip for a fiber-reinforced brittle matrix composite undergoing longitudinal tensile loading

Fig. 7 Crack Spacing as a Function of Applied Stress for Various Interface Toughnesses

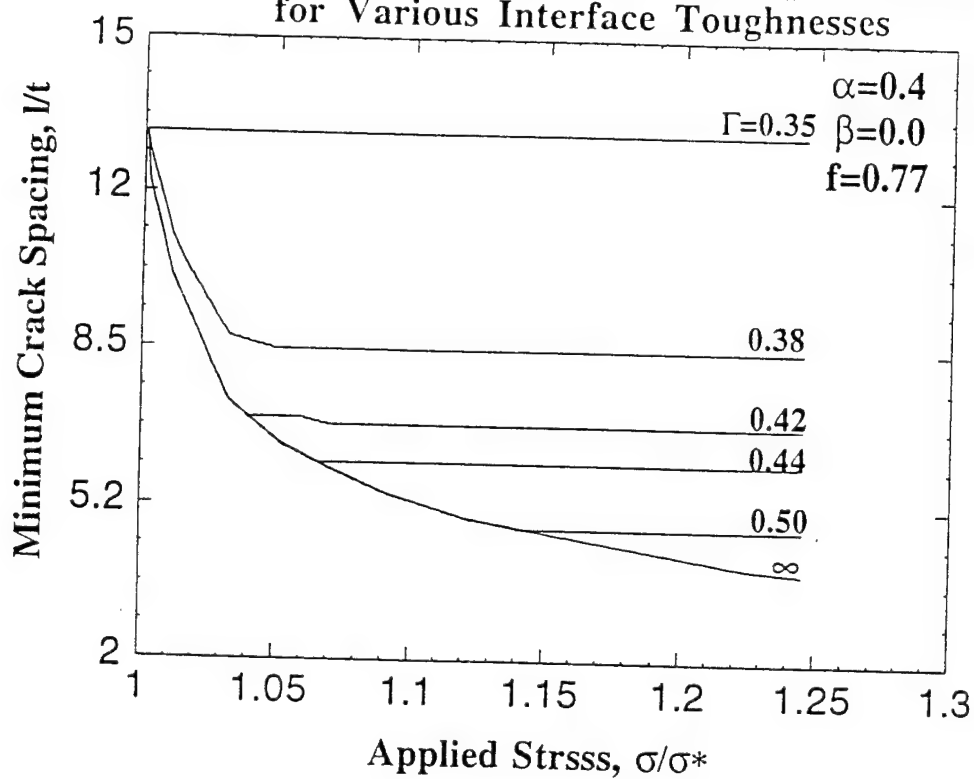
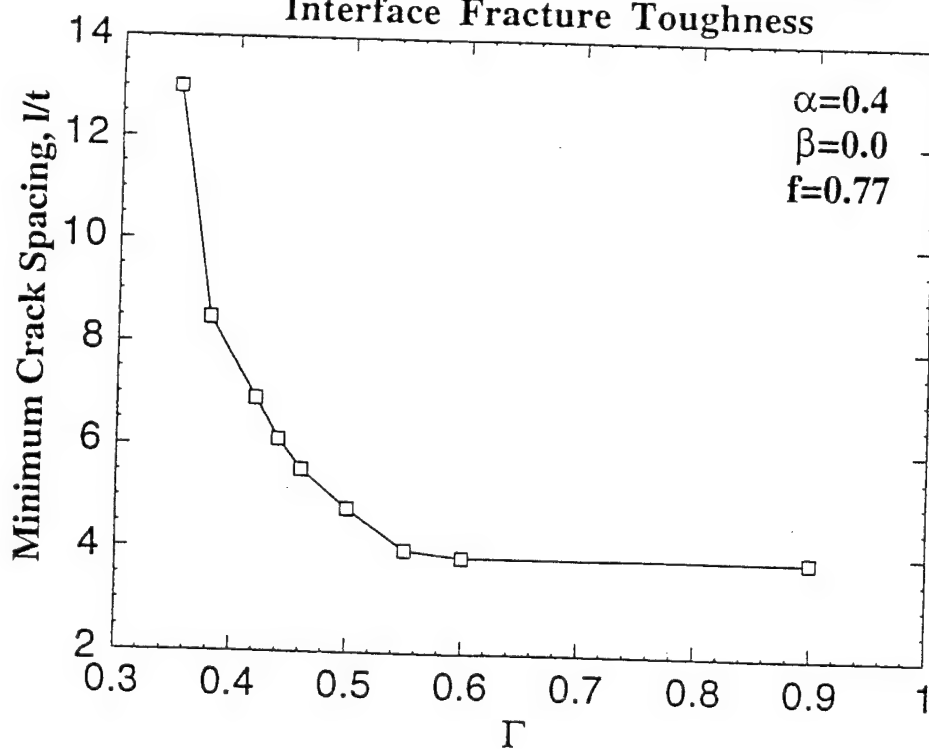
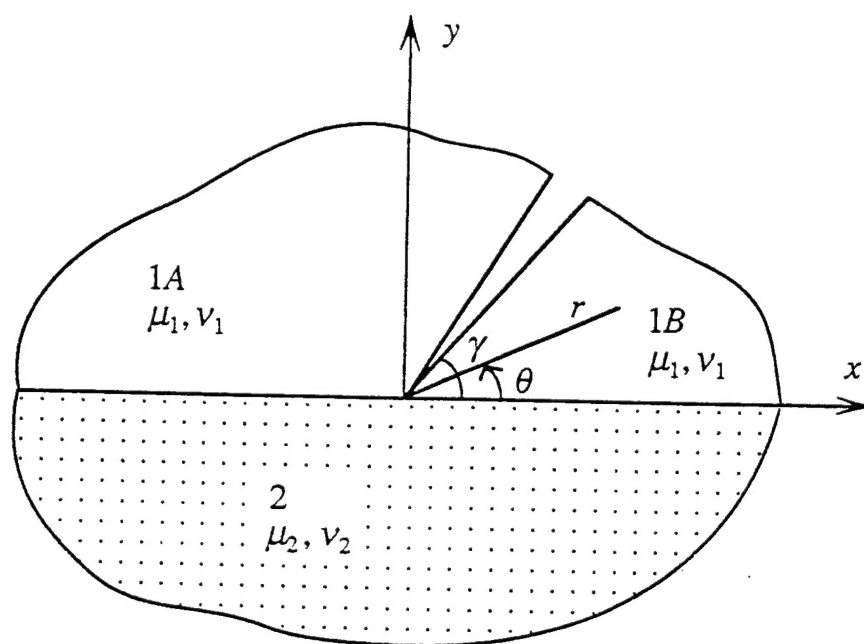


Fig. 8 Minimum Crack Spacing as a Function of Interface Fracture Toughness





**Fig. 9** A crack terminating at a frictional material interface

## 7. Presentations

J. Weertman, "Asymptotic Dislocation Density Fields for Cracks in an Elastic Perfectly Plastic Solid," International Conference on Fundamentals of Fracture (ICFF IV), Urbandai, Japan, May 31-June 4, 1993 (invited)

B. Moran, "Analysis of Multiple Cracking Configurations for Interface Strength and Toughness," symposium on Physics and Mechanics of Bond Strength at Interfaces, ASME Summer Annual Meeting, Charlottesville, VA, 1993.

A. Cazzato and K. T. Faber, "Cracking and Debonding of a Brittle Film on a Ductile Rod," Annual Meeting of the American Ceramic Society, Cincinnati, OH, April 20, 1993.

A. Cazzato and K.T. Faber, "Testing of Alumina-Sapphire Bimaterial Bend Specimen to Determine Interfacial Fracture Toughness," Annual Meeting of the American Ceramic Society, Indianapolis, IN, April 26, 1994.



## 8. Publications

Cazzato, A. and Faber, K.T. (1993) "Multiple Cracking and Debonding of a Brittle Film on a Ductile Rod," *Ceramic Transactions* - in press.

Forland, K.A., Weertman, J. and Faber, K.T. (1992) "Crack Tip Shielding/Antishielding by Precipitates in Silicon Carbide" (Abstract) American Ceramic Society Annual Meeting, Minneapolis, MN, April 12-16, 1992.

Forland, K.A., Faber, K.T., and Weertman, J. (1994) "Load Application at High Temperature," manuscript in preparation.

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Keer, L. M. and Polonsky, I. A. (1993) "The Displacement Field of a Triangular Phase Boundary Dislocation Loop," *Phil. Mag A*, Vol. 67, No. 6, pp. 1423-1431.

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Weertman, J. (1993) "Modes I and II Cracks in a Linear Hardening Solid in General Yielding," *Mechanics of Materials*, **15**, pp. 75-86.

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